PROCESS TO CONTINUOUSLY PREPARE TWO OR MORE BASE OIL
GRADES AND MIDDLE DISTILLATES

Field of invention

The invention relates to a process to continuously
prepare two or more base oil grades and middle
distillates from a mineral crude derived feed.

Background of the invention

WO-A-0250213 describes a process wherein different
base oil grades are made in a so-called blocked-out mode.
In this process a bottoms fraction of a hydrocracking
process, which thus also yields middle distillates as
products, is separated into various base oil precursor
fractions. These fractions are subsequently catalytically
dewaxed one after the other using a platinum-ZSM-5 based
catalyst.

WO-A-9718278 discloses a process wherein up to 4 base
oil grades, e.g. a 60N, 100N and 150N, are prepared
starting from the bottoms fraction of a hydrocracker. In
this process the bottoms fraction is first fractionated
in a vacuum distillation into 5 fractions of which the
heavier 4 fractions are further processed to different
base oil grades by first performing a catalytic dewaxing
followed by a hydrofinishing step.

A disadvantage of the above processes is that the
process is not continuous. In other words the base oil
grades are not made at the same time but sequential. This
requires tankage for the intermediate products as
obtained when the hydrocracker bottoms are fractionated
and are awaiting their turn to be catalytically dewaxed.
A further disadvantage are the mode switches which result
in heating up of equipment and cooling of equipment
causing deterionation of equipment. The mode switches

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also result in intermediate off-spec product every time a new grade is being processed. These slops need to be reprocessed or disposed off which is disadvantageous.

EP-A-649896 discloses a process to prepare a residue comprising a base oil by means of a process involving a hydrotreating step and a hydrocracking step on a heavy petroleum feedstock. The hydroprocessing steps yield a product from which middle distillates and a bottoms fraction (residue) are obtained. This bottoms fraction is subsequently solvent dewaxed to a single grade base oil grade.

WO 02/070627 discloses a process to prepare two or more base oil grades and a gas oil from a Fischer-Tropsch derived feed, i.e. not from a mineral crude derived feed.

EP-A-0 994 173 relates to a process for making an automatic transmission fluid composition.

US-A-6 576 120 relates to a process for the catalytic dewaxing of a hydrocarbon feed containing waxy molecules.

WO-A-9723584 discloses a process wherein the bottoms fraction of a fuels hydrocracker is subjected to a catalytic dewaxing step. The dewaxed oil is partly recycled to the hydrocracking step and partly obtained as the lubricating base oil.

A disadvantage of the above process as described in WO-A-9723584 is that if more than one base oil grade is isolated from the dewaxed oil a large pour point distribution occurs. In other words the resulting the lower viscous base oil grades will have a too low pour point. This pour point give away, or difference with the desired value, is indicative for yield loss of said lower viscous base oil grade.

One object of the present invention is to provide a process, which is capable of preparing two or more base oil grades simultaneously and wherein their respective pour points are more close to the desired values.

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It is another object of the present invention to provide an alternative process to prepare two or more base oil grades simultaneously from a mineral crude derived feed.

5   Summary of the invention

One or more of the above or other objects is achieved according to the present invention by providing the following process.

Process to prepare simultaneously two or more base
10   oil grades and middle distillates from a mineral crude derived feed, in particular a de-asphalted oil or a vacuum distillate feed or their mixtures, by performing the following steps:

(a) hydrocracking the mineral crude derived feed, thereby
15   obtaining an effluent;

(b) distillation of the effluent as obtained in step (a) into one or more middle distillates and a full range residue boiling substantially above 340 °C,

(c) catalytically dewaxing the full range residue by
20   contacting the residue with a dewaxing catalyst comprising a zeolite of the MTW type and a Group VIII metal, thereby obtaining a dewaxed oil;

(d) isolating by means of distillation two or more base oil grades from the dewaxed oil obtained in step (c); and

25   (e) isolating a dewaxed gas oil from the dewaxed-oil obtained in step (c);

wherein the dewaxed oil as obtained in step (c) comprises between 10 and 40 wt% of a dewaxed heavy gas oil boiling for more than 70 wt% between 370 and 400 °C.

30   Applicants found that when using a catalytic dewaxing catalyst comprising a MTW zeolite type a more equally distributed pour point value was achieved for all the base oil grades as obtained in step (d). Because of this more equal pour point quality of the resulting grades a
35   minimal quality give away will result, which makes this

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mode of operation attractive. If in contrast a ZSM-5
based dewaxing catalyst was used on such a full range
residue as described in WO-A-9723584 a much greater pour
point distribution will be observed. In case such a
5   catalyst would be used the lower viscosity grades would
have a pour point much lower than required in order to
achieve the desired pour point in the remaining higher
viscosity base oil grades. The lower than required pour
point is a disadvantage because it is indicative for a
10   lower yield of said lower viscosity grade base oil.

Another advantage of processing the full range
residue in comparison to the blocked-out mode is that any
compounds which are converted in the more heavier base
oil grades to compounds boiling just below said grade
15   contribute to the base oil yield just boiling below said
heavier grade. In case of a blocked-out mode of operation
these compounds boiling just below the desired grade
cannot be easily combined with the less viscous base oil.

An even further advantage is that the Applicants
20   surprisingly found that a dewaxing catalyst comprising a
zeolite of the MTW type and a Group VIII metal provides
not only two or more base oil grades having desirable
properties, but also surprisingly high amounts of a heavy
gas oil having a surprisingly low pour point.
25   Detailed description of the invention.

The feed to step (a) may be any typical mineral crude
derived feed to a hydrocracker. Such feedstocks may be
the vacuum gas oil or heavier distillate fractions as
obtained when distilling at vacuum conditions the
30   atmospheric residue of a crude mineral oil feedstock. The
deasphalted oil as obtained when deasphalting the residue
as obtained in said vacuum distillation may also be used
as feed. Light and heavy cycle oils as obtained in a
fluid catalytic cracking process (FCC), thermally flashed
35   distillate and aromatic rich extracts as for example

obtained in solvent extraction process steps in traditional base oil processing may also be used as feed. Mixtures of the above described feeds and optionally other hydrocarbon sources are also suitable as feedstocks. An optional alternative hydrocarbon source which may be blended with the mineral source feedstocks as described above are the, optionally partly isomerised, paraffin waxes as obtained in a Fischer-Tropsch process. The amount of such paraffin wax is preferably not more than 30 wt% in the feed to step (a). Preferably, the feed is 100% mineral crude derived.

In order to be able to prepare the desired quantity of the more viscous base oil grades a relatively heavy feed to step (a) is desired. Preferably a feed is used wherein more than 10 wt%, preferably more than 20 wt% and most preferably more than 30 wt% of the compounds present in said feed boil above 470 °C. Suitably less than 60 wt% of the compounds present in the feed boil above 470 °C.

Step (a) may be performed at a conversion level of between 15 and 90 wt%. The conversion is expressed in the weight percentage of the fraction in the feed which boils above 370 °C which are converted to products boiling below 370 °C. The main products boiling below 370 °C are naphtha, kerosene and gas oil. Examples of possible hydrocracker processes suitable for performing step (a) are described in EP-A-699225, EP-A-649896, WO-A-9718278, EP-A-705321, EP-A-994173 and US-A-4851109.

The operating conditions of a single step hydrocracking process are preferably a temperature in the range of from 350 to 450 °C, a hydrogen pressures in the range of from 9 to 200 MPa, more preferably above 11 MPa, a weight hourly space velocities (WHSV) in the range of from 0.1 to 10 kg of oil per liter of catalyst per hour (kg/l/hr), preferably from 0.2 to 5 kg/l/hr, more preferably from 0.5 to 3 kg/l/hr and hydrogen to oil

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ratios in the range of from 100 to 2,000 liters of
hydrogen per liter of oil.

Preferably the hydrocracker is operated in two steps,
consisting of a preliminary hydrotreating step followed
5   by a hydrocracking step. In the hydrotreating step
nitrogen and sulphur are removed and aromatics are
saturated to naphthenes and part of the naphthenes are
converted to paraffins by ring opening reactions. In
order to improve the yield of the more viscous grade base
10   oils the hydrocracker is more preferably operated by
first (i) hydrotreating a hydrocarbon feed at a feed
conversion, wherein the conversion, as defined above, of
less than 30 wt% and preferably between 5 and 25 wt%, and
(ii) hydrocracking the product of step (i) in the
15   presence of a hydrocracking catalyst at such a conversion
level that the overall conversion of step (i) and (ii) is
between 15 and 90 wt% and preferably between 40 and
85 wt%.

The operating conditions of a hydrotreating step are
20   preferably a temperature in the range of from 350 to
450 °C, a hydrogen pressures in the range of from 9 to
200 MPa, more preferably above 11 MPa, a weight hourly
space velocities (WHSV) in the range of from 0.1 to 10 kg
of oil per liter of catalyst per hour (kg/l/hr),
25   preferably from 0.2 to 5 kg/l/hr, more preferably from
0.5 to 3 kg/l/hr and hydrogen to oil ratios in the range
of from 100 to 2,000 liters of hydrogen per liter of oil.

The operating conditions of a hydrocracking step
performed in combination with a hydrotreating step are
30   preferably a temperature in the range of from 300 to
450 °C, a hydrogen pressures in the range of from 9 to
200 MPa, more preferably above 11 MPa, a weight hourly
space velocities (WHSV) in the range of from 0.1 to 10 kg
of oil per liter of catalyst per hour (kg/l/hr),
35   preferably from 0.2 to 5 kg/l/hr, more preferably from

0.5 to 3 kg/l/hr and hydrogen to oil ratios in the range
of from 100 to 2,000 liters of hydrogen per liter of oil.

The full range residue as prepared by the processes
as described above have a very low content of sulphur,
typically below 250 ppm or even below 150 ppmw, and a
very low content of nitrogen, typically below 30 ppmw.

It has been found that by performing the combined
hydrotreating and hydrocracking step as described above a
full range residue is obtained which yields a high
quantity of the more viscous grade base oil, also
referred to as medium machine oil grade, and of
acceptable quality with respect to viscosity index. In
addition a sufficient quantity of naphtha, kerosine and
gas oils are obtained by this process. Thus a
hydrocracker process is obtained wherein simultaneously
products ranging from naphtha to gas oil and a full range
residue is obtained, which full range residue has the
potential to yield a medium machine oil base oil grade.
The viscosity index of the resulting base oil grades is
suitably between 95 and 120, which is acceptable to yield
base oils having a viscosity index according to the API
Group II specifications. It has been found that the wt%
of medium machine oil fraction, which fraction has a
kinematic viscosity at 100 °C of above 9 cSt, in the
370 °C plus fraction of the full range residue can be
more than 15 wt% and more especially more than 25 wt% if
the hydrocracker is operated as described above.

In the context of the present invention terms as
spindle oil, light machine oil and medium machine oil
will refer to base oil grades having an increasing
kinematic viscosity at 100 °C and wherein the spindle oil
additionally has a maximum volatility specification. The
advantages of the present process are achieved for any
group of base oils having such different viscosity
requirement and volatility specification. Preferably a

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spindle oil is a light base oil product having a
kinematic viscosity at 100 °C of below 5.5 cSt and
preferably above 3.5. The spindle oil can have either a
Noack volatility, as determined by the CEC L-40-T87
method, of preferably below 20% and more preferably below
18% or a flash point, as measured according to ASTM D93,
of above 180 °C. Preferably the light machine oil has a
kinematic viscosity at 100 °C of below 9 cSt and
preferably above 6.5 cSt and more preferably between 8
and 9 cSt. Preferably the medium machine oil has a
kinematic viscosity at 100 °C of below 14 cSt and
preferably above 10 cSt and more preferably between 11
and 13 cSt. These base oil grades preferably have a
viscosity index of between 95 and 120.

It has been found that in the hydrotreating step (i)
the viscosity index of the full range residue and the
resulting base oil grades increases with the conversion
in said hydrotreating step. By operating the
hydrotreating step at high conversion levels of more than
30 wt% viscosity index values for the resulting base oils
of well above 120 can be achieved. A disadvantage of such
a high conversion in step (i) is however that the yield
of medium machine oil fraction will be undesirably low.
By performing step (i) at the above described conversion
levels an API Group II medium machine oil grade base oil
can be obtained in a desired quantity. The minimum
conversion in step (i) will be determined by the desired
viscosity index, of between 95 and 120, of the resulting
base oil grades and the maximum conversion in step (i) is
determined by the minimum acceptable yield of medium
machine oil grade.

The preliminary hydrotreating step is typically
performed using catalyst and conditions as for example
described in the above-mentioned publications related to
hydrocracking. Suitable hydrotreating catalysts generally

comprise a metal hydrogenation component, suitably
Group IVB or VIII metal, for example cobalt-molybdenum,
nickel-molybdenum, on a porous support, for example
silica-alumina or alumina. The hydrotreating catalysts
suitably contains no zeolite material or a very low
content of less than 1 wt%. Examples of suitable
hydrotreating catalysts are the commercial ICR 106,
ICR 120 of Chevron Research and Technology Co.; 244, 411,
DN3100, DN3110, DN3120, DN3300, DN120, DN190 and DN200 of
Criterion Catalyst Co.; TK-555 and TK-565 of Haldor
Topsoe A/S; HC-k, HC-P, HC-R and HC-T of UOP; KF-742,
KF-752, KF-846, KF-848 STARS and KF-849 of AKZO
Nobel/Nippon Ketjen; and HR-438/448 of Procatalyse SA.

The hydrocracking step is preferably a catalyst
comprising an acidic large pore size zeolite within a
porous support material with an added metal hydro-
genation/dehydrogenation function. The metal having the
hydrogenation/dehydrogenation function is preferably a
Group VIII/Group VIB metal combination, for example
nickel-molybdenum and nickel-tungsten. The support is
preferably a porous support, for example silica-alumina
and alumina. It has been found that a minimum amount of
zeolite is advantageously present in the catalyst in
order to obtain a high yield of medium machine oil
fraction in the full range residue when performing the
hydrocracker at the preferred conversion levels as
explained above. Preferably more than 1 wt% of zeolite is
present in the catalyst. Examples of suitable zeolites
are zeolite X, Y, ZSM-3, ZSM-18, ZSM-20 and zeolite beta
of which zeolite Y is most preferred. Examples of
suitable hydrocracking catalysts are the commercial
ICR 220 and ICR 142 of Chevron Research and Technology
Co; Z-763, Z-863, Z-753, Z-703, Z-803, Z-733, Z-723,
Z-673, Z-603 and Z-623 of Zeolyst International; TK-931
of Haldor Topsoe A/S; DHC-32, DHC-41, HC-24, HC-26, HC-34

and HC-43 of UOP; KC2600/1, KC2602, KC2610, KC2702 and
KC2710 of AKZO Nobel/Nippon Ketjen; and HYC 642 and
HYC 652 of Procatalyse SA.

The effluent of the hydrocracker is separated into
one or more of the above referred to fuels fractions and
a full range residue. The full range residue is defined
as the bottoms product of the distillation of the
effluent of step (a) at atmospheric conditions.
Preferably the full range residue boils predominately
above 340 °C. With boiling predominately above 340 °C is
especially meant that more than 80 wt% boils above
340 °C. Because a substantial fraction of the full range
residue may boil in the gas oil range a considerable
amount of gas oil is recovered after dewaxing having a
excellent cold flow properties. It has also been found
that when the MTW based catalyst is used in step (c) a
relatively high selectivity is achieved to gas oil in
comparison to when other dewaxing catalyst are used. This
is one of the key advantages of the present invention.
According to the present invention between 10 and 40 wt%
of the dewaxed oil boils in the heavy gas oil range being
between from 350 to 400 °C. It must be understood that
the dewaxed oil also comprises lower boiling gas oil
components.

The final boiling point of the residue will be partly
determined by the final boiling point of the feed to
step (a) and may be far greater than 700 °C up to values
of which cannot be determined by means of the standard
test methods.

Thus, the full range residue as obtained in step (b)
and used as feed to step (c) will not have been subjected
to a distillation step wherein compounds boiling above
420 °C have been separated from the residue. This is
advantageous because it eliminates the need for such a
distillation unit in this part of the process.

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Optionally part of the full range residue as obtained in step (a) may be recycled to step (a) as for example described in EP-A-0994173, which publication is incorporated by reference. Optionally the residue may be recycled to only the hydrocracking step of step (a) as for example described in EP-B-0699225, which publication is incorporated by reference. Preferably less than 15 wt% of the residue is recycled to step (a) and more preferably no residue is recycled to step (a). It has been found that good quality base oils may be prepared having a good quality without having to perform such a recycle. The feed to step (c) comprises the full range residue as obtained in step (b) and optionally a partly isomerised paraffin wax, also referred to as waxy raffinate, as obtained in a Fischer-Tropsch or Gas-to-Liquids Process. Such a waxy Raffinate may be prepared according the process as described in WO-02070630, which publication is incorporated herein by reference. Addition of such a Waxy Raffinate stream is advantageous to increase the viscosity index of the base oils in situations wherein the mineral source feed to step (a) is too poor to yield the desired viscosity index of the base oil grades. Adding Waxy Raffinate is also advantageous because the potential viscosity index of the residue may be lower thereby allowing less severe hydroprocessing conditions, i.e. at lower conversions, in step (a). This in turn will result in a higher base oil yield on mineral source feed to step (a). Up to 60 wt% of the feed to step (c) may advantageously comprise of this waxy Raffinate.

Optionally a noble metal guard bed may be positioned just upstream the dewaxing catalyst bed in the dewaxing reactor in order to remove any remaining sulphur and especially nitrogen compounds. An example of such a

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process is described in WO-A-9802503, which reference is
hereby incorporated by reference.

The catalyst composition of the catalyst used in
step (c) comprises a Group VIII metal and a zeolite of
5   the MTW type. Preferably the catalyst also comprises of a
binder.

Examples of MTW type zeolites are ZSM-12 as described
in US-A-3,832,449, CZH-5 as described in GB-A-2079735,
Gallosilicate MTW as described in Y.X. Zhi, A. Tuel,
10   Y. Bentaarit and C. Naccache, Zeolites 12, 138 (1992),
Nu-13(5) as described in EP-A-59059, Theta-3 as described
in EP-A-162719, TPZ-12 as described in US-A-4557919 and
VS-12 as described in K. M. Reddy, I. Moudrakovski and A.
Sayari, J. Chem. Soc., Chem. Commun. 1994, 1491 (1994).
15   The average crystal size of the zeolite is preferably
smaller than 0.5 μm and more preferably smaller than
0.1 μm as determined by the well-known X-ray diffraction
(XRD) line broadening technique using the high intensity
peak at about 20.9 2-theta in the XRD diffraction
20   pattern.

The binder in the catalyst may be any binder usually
used for such an application. A possible binder includes
alumina or alumina containing binders. Applicants have
found that low acidity refractory oxide binder material
25   that is essentially free of alumina provides more
improved catalyst. Examples are low acidity refractory
oxides such as silica, zirconia, titanium dioxide,
germanium dioxide, boria and mixtures of two or more of
these. The most preferred binder is silica. The weight
30   ratio of the molecular sieve and the binder can be
anywhere between 5:95 and 95:5. Lower zeolite content,
suitable between 5 and 35 wt%, is advantageous for
achieving an even higher selectivity.

The silica to alumina molar ratio of the zeolite
35   prior to dealumination is preferably larger than 50 and

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more preferably between 70 and 250 and most preferably
between 70 and 150. Preferably the zeolite has been
subjected to a dealumination treatment. The dealumination
of the zeolite results in a reduction of the number of
5   alumina moieties present in the zeolite and hence in a
reduction of the mole percentage of alumina. The
expression "alumina moiety" as used in this connection
refers to an Al_2O_3-unit which is part of the framework of
the aluminosilicate zeolite, i.e. which has been
10   incorporated via covalent bindings with other oxide
moieties, such as silica (SiO_2), in the framework of the
zeolite. The mole percentage of alumina present in the
aluminosilicate zeolite is defined as the percentage of
moles Al_2O_3 relative to the total number of moles of
15   oxides constituting the aluminosilicate zeolite (prior to
dealumination) or modified molecular sieve (after
dealumination). Preferably dealumination is performed
such that the reduction in alumina moieties in the
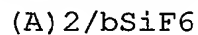framework is between 0.1 and 20%.
20   Dealumination may be performed by means of steaming.
Preferably the surface of the zeolite crystallites are
selectively dealuminated. A selective surface
dealumination results in a reduction of the number of
surface acid sites of the zeolite crystallites, whilst
25   not affecting the internal structure of the zeolite
crystallites. When applying a surface dealumination the
reduction of alumina moieties in the framework will be
lower and preferably between 0.1 and 10%. Dealumination
using steam results is a typical non-selective
30   dealumination technique.
Dealumination can be attained by methods known in the
art. Particularly useful methods are those, wherein the
dealumination selectively occurs, or anyhow is claimed to
occur selectively, at the surface of the crystallites of

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the molecular sieve. Examples of dealumination processes
are described in WO-A-9641849. US-A-5015361 describes a
method wherein the zeolites are contacted with sterically
hindered amine compound.

Preferably dealumination is performed by a process in
which the zeolite is contacted with an aqueous solution
of a fluorosilicate salt wherein the fluorosilicate salt
is represented by the formula:

$$(A)2/bSiF6$$

wherein 'A' is a metallic or non-metallic cation other
than H+ having the valence 'b'. Examples of cations 'b'
are alkylammonium, NH4+, Mg++, Li+, Na+, K+, Ba++, Cd++,
Cu+, Ca++, Cs+, Fe++, Co++, Pb++, Mn++, Rb+, Ag+, Sr++,
Tl+, and Zn++. Preferably 'A' is the ammonium cation.
The zeolite material may be contacted with the
fluorosilicate salt at a pH of suitably between 3 and 7.
Such a dealumination process is for example described
in US-A-5157191. The dealumination treatment is also
referred to as the AHS-treatment.

The catalyst composition is preferably prepared by
first extruding the zeolite with the low acidity binder
and subsequently subjecting the extrudate to a
dealumination treatment, preferably the AHS treatment as
described above. It has been found that an increased
mechanical strength of the catalyst extrudate is obtained
when prepared according to this sequence of steps.

It is believed that by maintaining the acidity of the
catalyst at a low level conversion to products boiling
outside the lube boiling range is reduced. Applicants
found that the catalyst should have an alpha value below
50 prior to metals addition, preferably below 30, and
more preferably below 10. The alpha value is an
approximate indication of the catalytic cracking activity
of the catalyst compared to a standard catalyst. The
alpha test gives the relative rate constant (rate of

normal hexane conversion per volume of catalyst per unit
time) of the test catalyst relative to the standard
catalyst which is taken as an alpha of 1 (Rate
Constant=0.016 sec -1). The alpha test is described in

5   U.S. Pat. No. 3,354,078 and in J. Catalysis, 4,
527 (1965); 6, 278 (1966); and 61, 395 (1980), to which
reference is made for a description of the test.
The experimental conditions of the test used to determine
the alpha values referred to in this specification

10   include a constant temperature of 538° C. and a variable
flow rate as described in detail in J. Catalysis, 61,
395 (1980).

The Group VIII metal may be nickel or cobalt or more
preferably a noble metal Group VIII metal. Preferred

15   noble Group VIII metals are palladium and more preferably
platinum. The total amount platinum or palladium will
suitably not exceed 10% by weight calculated as element
and based on total weight of the catalyst, and preferably
is in the range of from 0.1 to 5.0% by weight, more

20   preferably from 0.2 to 3.0% by weight. If both platinum
and palladium are present, the weight ratio of platinum
to palladium may vary within wide limits, but suitably is
in the range of from 0.05 to 10, more suitably 0.1 to 5.
Catalysts comprising palladium and/or platinum as the

25   hydrogenation component are preferred. Most preferred is
when platinum is used as the sole hydrogenation
component. The hydrogenation component is suitably added
to the catalyst extrudate comprising the dealuminated
aluminosilicate zeolite crystallites by known techniques.

30   The process conditions used in step (c) are
preferably typical catalytic dewaxing process conditions
involving operating temperatures in the range of from 200
to 500 °C, preferably from 250 to 400 °C, more preferably
between 300 and 380 °C, hydrogen partial pressures in the

35   range of from 10 to 200 bar preferably from 30 to

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150 bar, more preferably from 30 to 60 bar. Weight hourly space velocities (WHSV) in the range of from 0.1 to 10 kg of oil per liter of catalyst per hour (kg/l/hr), preferably from 0.2 to 5 kg/l/hr, more preferably from
5   0.5 to 3 kg/l/hr and hydrogen to oil ratios in the range of from 100 to 2,000 liters of hydrogen per liter of oil. The pour point of the base oil grades will vary depending their viscosity. The pour point of the various grades will also be dependent on the dewaxing severity in
10   step (c) and will preferably be between −40 and −10 °C.

Prior to performing step (d) the effluent or part of the effluent of step (c) is preferably subjected to a hydrofinishing step (c2). Preferably the entire effluent of step (c) is subjected to a hydrofinishing step.
15   Alternatively one may separate the lower viscous grades, especially the gas oil and optionally also the spindle oil, from the effluent of step (c) and perform step (c2) exclusively on the fraction comprising the more viscous grades. It has been found that the lower viscous grades
20   do not always require a hydrofinishing step to obtain a water white oil or gas oil having the desired long term stability. Optionally the gas oil and the fractions boiling below said gas oil are topped from the effluent of step (c) in a simple distillation column. This is
25   advantageous because it enables one to process a full range residue comprising a larger portion of low boiling compounds, boiling in the (vacuum) gas oil range, without having to install for example a hydrofinishing reactor for these fractions. This because the extra gas oil
30   fraction in the effluent of step (c) is separated from said effluent before performing hydrofinishing step (c2).

The hydrofinishing step is to improve the quality of the dewaxed fraction. In this step lube range olefins are saturated, heteroatoms and colour bodies are removed and
35   if the pressure is high enough residual aromatics are

saturated. Preferably the conditions are so chosen to obtain a base oil grade comprising more than 95 wt% saturates and more preferably such that a base oil is obtained comprising more than 98 wt% saturates. The hydrofinishing step is suitable carried out at a temperature between 230 and 380 °C, a hydrogen partial pressure of between 10 to 250 bar and preferably above 100 bar and more preferably between 120 and 250 bar. The WHSV (Weight hourly space velocity) ranges from 0.3 to 10 kg of oil per litre of catalyst per hour (kg/l.h).

The hydrofinishing step is suitably carried out in cascade with the dewaxing step. Thus the entire effluent of step (c) is fed to step (c2), without separating any products between said steps as also described above. For this reason the operating hydrogen partial pressure in the dewaxing step is determined by the required hydrogen partial pressure of the hydrofinishing step and thus preferably above 100 bar and more preferably between 120 and 250 bar.

The hydrofinishing or hydrogenation catalyst is suitably a supported catalyst comprising a dispersed Group VIII metal. Possible Group VIII metals are cobalt, nickel, palladium and platinum. Cobalt and nickel containing catalysts may also comprise a Group VIB metal, suitably molybdenum and tungsten.

Suitable carrier or support materials are low acidity amorphous refractory oxides. Examples of suitable amorphous refractory oxides include inorganic oxides, such as alumina, silica, titania, zirconia, boria, silica-alumina, fluorided alumina, fluorided silica-alumina and mixtures of two or more of these.

Suitable hydrogenation catalysts include those catalysts comprising as one or more of nickel (Ni) and cobalt (Co) in an amount of from 1 to 25 percent by weight (wt%), preferably 2 to 15 wt%, calculated as

element relative to total weight of catalyst and as the Group VIB metal component one or more of in an amount of from 5 to 30 wt%, preferably 10 to 25 wt%, calculated as element relative to total weight of catalyst. Examples of suitable nickel-molybdenum containing catalyst are KF-847 and KF-8010 (AKZO Nobel) M-8-24 and M-8-25 (BASF), and C-424, DN-190, HDS-3 and HDS-4 (Criterion). Examples of suitable nickel-tungsten containing catalysts are NI-4342 and NI-4352 (Engelhard), C-454 (Criterion). Examples of suitable cobalt-molybdenum containing catalysts are KF-330 (AKZO-Nobel), HDS-22 (Criterion) and HPC-601 (Engelhard).

For hydrocracked feeds containing low amount of sulphur, as in the present invention, preferably platinum containing and more preferably platinum and palladium containing catalysts are used. The total amount of these noble Group VIII metal component(s) present on the catalyst is suitably from 0.1 to 10 wt%, preferably 0.2 to 5 wt%, which weight percentage indicates the amount of metal (calculated as element) relative to total weight of catalyst.

Preferred supports for these palladium and/or platinum containing catalysts are amorphous silica-alumina, whereby more preferably the silica-alumina comprises from 2 to 75 wt% of alumina. Examples of suitable silica-alumina carriers are disclosed in WO-A-9410263. A preferred catalyst comprises an alloy of palladium and platinum preferably supported on an amorphous silica-alumina carrier of which the commercially available catalysts C-624 and C-654 of Criterion Catalyst Company (Houston, TX) are examples.

Step (d) and (e) may be performed in one or more distillation columns operating at a reduced pressure. As explained above distillation may be performed in two

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stages wherein only the heavier part is subjected to a
hydrofinishing step.

The different base oil grades may be obtained by
withdrawing products along the distillation column,
preferably using so-called side-strippers. Intermediate
fractions may also be withdrawn in order to meet the
volatility requirements of the desired base oil grades.
Preferably gaseous tops, a liquid tops, a spindle oil, a
light machine oil and a medium machine oil are obtained
in step (d) and (e).

The liquid tops, boiling preferably and substantially
below 400 °C as obtained in step (d) and (e) comprises
naphtha, kerosene and gas oil fractions may be
advantageously recycled to step (b) or be isolated as
separate products. Because these products have been
subjected to a catalytic dewaxing step and to an optional
hydrofinishing step a fuel product is obtained having a
very low content of aromatics, sulphur together with
excellent low temperature properties. Especially a gas
oil may be obtained in step (d) and (e) having a very low
sulphur content of below 10 ppm, a low aromatics content
of below 0.1 mmol/100 grams, excellent a cold flow
properties like a pour point of below -30 °C and a cold
filter plugging point of below -30 °C. The gas oil also
has excellent lubricity properties. This makes such a gas
oil especially an excellent refinery blending component
to blend low sulphur gas oil. The gas oil may also be
used as a drilling mud fluid, an electrical oil, a
cutting oil, an aluminium rolling oil or as a fruit spray
oil.

The invention will be illustrated by the following
non-limiting examples.

Preparation of the dewaxing catalyst

MTW Type zeolite crystallites were prepared as
described in "Verified synthesis of zeolitic materials"

as published in Micropores and mesopores materials,
volume 22 (1998), pages 644-645 using tetra ethyl
ammonium bromide as the template. The Scanning Electron
Microscope (SEM) visual observed particle size showed
5   ZSM-12 particles of between 1 and 10 µm. The average
crystallite size as determined by XRD line broadening
technique as described above was 0.05 µm. The
crystallites thus obtained were extruded with a silica
binder (25% by weight of zeolite, 75% by weight of silica
10   binder). The extrudates were dried at 120 °C and calcined
at 625 °C. A solution of $(NH4)2SiF6$ (20 ml of 0.02 N
solution per gram of zeolite crystallites) was poured
onto the extrudates. The mixture was then heated at 90 °C
during 5 hours. After filtration, the extrudates were
15   washed twice with deionised water, dried at 120 °C and
then calcined at 500 °C.

The thus obtained extrudate was impregnated with an
aqueous solution of platinum tetramine nitrate and drying
and calcining in a rotary kiln at 300 °C. The resulting
20   catalyst comprised 0.7% by weight Pt supported on the
dealuminated, silica-bound MTW zeolite.

The catalyst was activated in-situ by reduction of
the platinum under a hydrogen rate of 100 l/hr at a
temperature of 350 °C for 2 hours.

25   Example 1

A full range residue as obtained in a hydrocracking
process, having the properties as listed in Table 1 was
contacted with the silica bound Pt-MTW catalyst as
described above in the presence of hydrogen at three
30   different temperatures as specified in Table 2, an outlet
pressure of 140 bar, a WHSV of 1 kg/l.hr and a hydrogen
gas rate of 750 Nl/kg feed.

The liquid effluent having a pour point of -19 °C was
separated into a heavy gas oil product and three base oil
35   grades of which properties are listed in Table 2.

Table 1

Full range residue		
Pour point		42 °C
Density at 70 °C		819.3
Refractive index at 70 °C		1.4530
Vk 100 °C (a)		4.786 cSt
S		< 1 mg/kg
N		< 1 mg/kg
Aromatics UV (SMS 2714 method)	mmol/100 g	
Monos	"	28,7
Naphthalenes	"	3,49
Phenantrenes	"	1,75
Chrysenes	"	0,589
Tetraphenes	"	1,242

(a) kinematic viscosity at 100 °C

TBP-GLC		
IBP	°C	182
10	"	341
50	"	434
90	"	528
95	"	552
99	"	589
FBP	"	600

Table 2

		HEAVY GAS OIL	SPINDLE OIL	LIGHT MACHINE OIL	MEDIUM MACHINE OIL
Distillation step		400 °C-	400°C-440°C	440°C-470°C	470°C+
%m on feed (from TBP GLC)			38,30	21,32	13,42	25,66
Pour point	°C	-35	-20	-14	-5
Vk 40 (b)	cSt	5,208	21,96	36,27	96,12
Vk 100 (b)	cSt	1,754	4,395	6,083	11,56
VI		nd	109	114	108

(b) kinematic viscosity at 40 °C or 100 °C resp.

Example 2

Example 1 was repeated except at a reactor temperature of 347 °C. The liquid effluent having a pour point of -29 °C was separated into a heavy gas oil
5   product and three base oil grades of which properties are listed in Table 3.

Table 3

		HEAVY GAS OIL	SPINDLE OIL	LIGHT MACHINE OIL	MEDIUM MACHINE OIL
Distillation step		400 °C-	400 °C-440 °C	440 °C-470 °C	470 °C+
%m on feed (from TBP GLC)			41,10	19,87	12,47	23,96
Pour point	°C	-39	-32	-25	-22
Vk 40	cSt	4,523	22,96	35,56	95,76
Vk 100	cSt	1,602	4,462	5,955	11,34
VI		nd	105	111	105

Example 3

Example 1 was repeated except at a reactor temperature of 350 °C. The liquid effluent having a pour point of −38 °C was separated into a heavy gas oil product and three base oil grades of which properties are listed in Table 4

Table 4

		HEAVY GAS OIL	SPINDLE OIL	LIGHT MACHINE OIL	MEDIUM MACHINE OIL
Distillation step		400 °C minus	400°C−440°C	440°C−470°C	470°C+
%m on feed (from TBP GLC)		23,96	18,82	11,74	22,80
Pour point	°C	−51	−38	−35	−24
Vk 40	cSt	3,884	23,39	37,85	102,6
Vk 100	cSt	1,455	4,439	6,081	11,71
VI		nd	98	105	102

nd = not determined

Comparative example A

Experiment 1 was repeated except that a catalyst based on ZSM-5 was used. The results of this experiment are presented in Figure 1 (straight line). Also in Figure 1 the results as presented in Table 2 are presented. The X-axis represents the mid boiling point of the base oil grade. The open triangles are the results of Example 1, the open boxes are the results of Example 2 and the open diamonds are a result of Example 3. The Figure shows that for the lower viscosity grades the ZSM-5 based catalyst results in a pour point give-away. A more flat pour point profile is obtained for Examples 1-3 according the invention.